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State of Washington

Standard Operating Procedure EAP036, Version 1.2

Benthic Flux Chambers

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Purpose of this document

The Department of Ecology develops Standard Operating Procedures (SOPs) to document agency practices related to sampling, field and laboratory analysis, and other aspects of the agency's technical operations.

Washington State Department of Ecology

Environmental Assessment Program (EAP)

Standard Operating Procedure for Benthic Flux Chambers

Version 1.2

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EAP036

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Signatures on File

Please note that the Washington State Department of Ecology's Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical and administrative experts. Their primary purpose is for internal Ecology use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. Distribution of these SOPs does not constitute an endorsement of a particular procedure or method.

Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the Department of Ecology.

Although Ecology follows the SOP in most instances, there may be instances in which Ecology uses an alternative methodology, procedure, or process.

SOP Revision History

Revision Date	Rev number	Summary of changes	Sections	Reviser(s)
10/24/2007	1.0	Added footer, title page, disclaimer	all	Bill Kammin
08/09/2012	1.1	Minor revisions; three-year review	4, 6, 9, 10	Roberts, M.
08/04/15	1.2	Minor revisions; three-year review	4, 5, 6, 7, 10	Roberts, M.

1.0 Purpose and Scope

1.1 This document is the Environmental Assessment Program (EAP) Standard Operating Procedure (SOP) for deployment and operation of benthic flux chambers.

1.2 In marine and freshwaters, water quality often reflects the complex interaction with the sediments. The exchange of dissolved oxygen and nutrients, called fluxes, is necessary to understand the effect of all nutrient sources on water column dissolved oxygen levels. The term benthic flux chambers refers to equipment that isolates a portion of the water column nearest the sediment surface and allows for the quantification of dissolved oxygen and nutrient fluxes between the sediments and the lower water column.

2.0 Applicability

2.1 The procedure will be followed for studies quantifying the exchange of both dissolved oxygen and nutrients between sediments and the overlying water column.

3.0 Definitions

3.1	DO	Dissolved oxygen
3.2	EAP	Environmental Assessment Program of the Department of Ecology
3.3	OC	Operations Center, 8270 28th Court NE, Lacey, WA 98516
3.4	LAR	Lab Analysis Request
3.5	MSDS	Material Safety Data Sheets
3.6	QAPP	Quality Assurance Project Plan

4.0 Personnel Qualifications/Responsibilities

4.1 All field staff must comply with the requirements of the EAP Safety Manual (EA Program, 2015). A full working knowledge of the procedures in Chapter 1 "General Field Work" is expected. If chambers are deployed in freshwater, field staff must comply with the section, "Working in Rivers and Streams." If chambers are deployed in marine waters, field staff must comply with the Chapter 2 section, "Operating Winches on Small Boats, Trailers, and Vehicles;" all onboard staff must be familiar with Chapter 3, "Boating."

4.2 The Field Lead directing deployment and sample collection must be knowledgeable of all aspects of the project's Quality Assurance Project Plan (QAPP) to ensure that credible and usable data are collected. All field staff should be briefed by the Field Lead or Project Manager on the sampling goals and objectives prior to arriving at the site.

5.0 Equipment, Reagents, and Supplies

5.1 General Equipment and Supplies

5.1.1 Benthic flux chambers, with retrieval lines and floats for identification and handling and flexible tubing for sample collection (Figure 1).

5.1.2 Hydrolabs or other continuous DO monitoring equipment with mounts to benthic flux chambers.

5.1.3 Peristaltic pump and batteries, or similar equipment, for sample collection.

5.1.4 Safety equipment appropriate for the sampling sites: safety vests or boating safety equipment; latex or nitrile gloves for hygienic protection; and leather gloves for handling ropes and cables.

5.1.5 Coolers with ice (regular or blue).

5.1.6 Field notebook and pens.

5.1.7 Safety goggles (for adding reagents to dissolved oxygen samples).

5.1.8 Lab Analysis Required (LAR) forms.

5.1.9 Sample tags.

5.1.10 Alligator clips.

5.1.11 Nylon zip ties.

5.1.12 Shears for cutting nylon zip ties.

5.1.13 Container for purging stagnant water in tubing.

5.1.14 Carrying case for miscellaneous equipment.

5.1.15 Electrical tape.

5.1.16 Extra bags for waste.

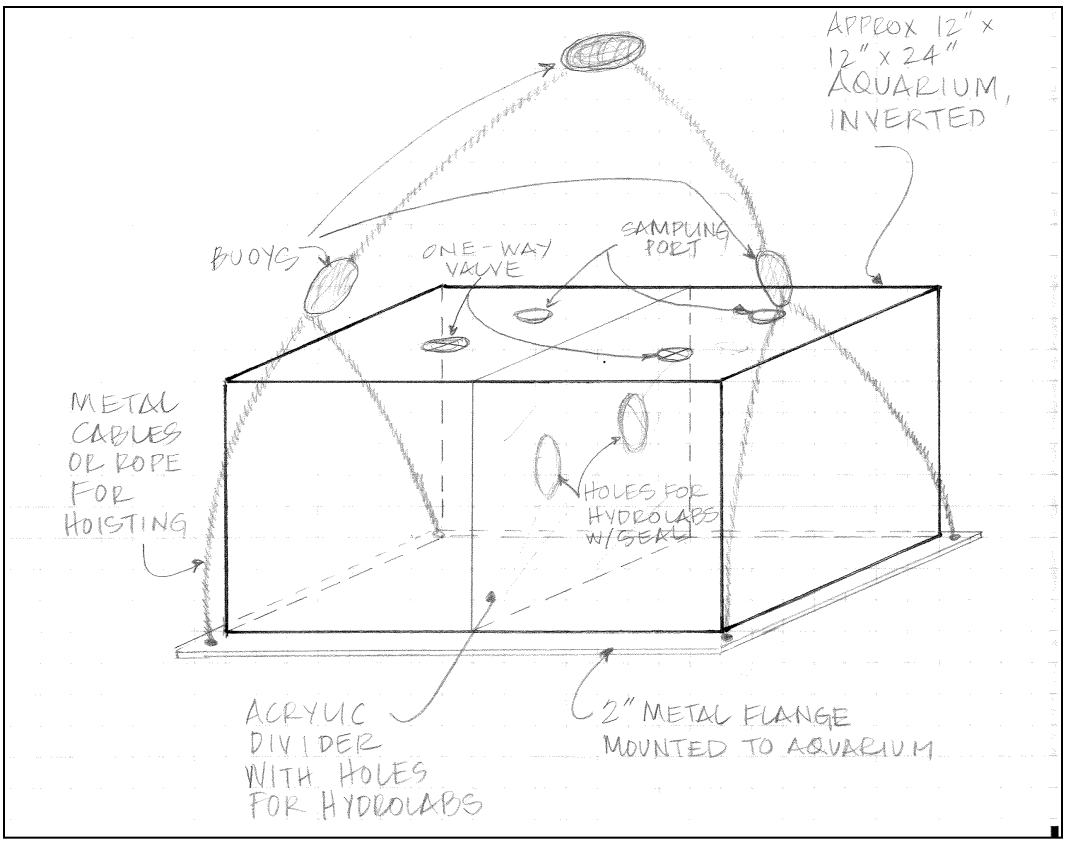

Figure 1. Schematic of benthic flux chamber, consisting of an inverted box with the open side toward the sediment.

5.2 Sample Containers

5.2.1 DO Samples

5.2.1.1 125-mL glass flasks and stoppers for grab sample analyses of dissolved oxygen concentrations using a Winkler titration.

5.2.1.2 See SOP EAP023 (Ward and Mathieu, 2013) for reagents, MSDS, materials, and solutions.

5.2.1.3 Deionized water.

5.2.2 Nitrogen and Phosphorus Samples

5.2.2.1 Large polypropylene bottles, 1 L, that have been acid washed and rinsed, if using an interim container for nutrient sample collection.

5.2.2.2 Total nitrogen and phosphorus sample bottles (60-mL polypropylene), acid washed and rinsed.

5.2.2.3 Dissolved nitrogen and phosphorus sample bottles (175-mL square polypropylene), acid washed and rinsed.

5.2.2.4 60-mL plastic syringes, obtained from the laboratory.

5.2.2.5 20-mL graduated cylinders, obtained from the laboratory, if not using syringe to measure total nitrogen and phosphorus sample.

5.2.2.6 25-mm, 0.45-micron disk filters for the syringes, obtained from the laboratory.

5.2.3 Carbon Samples

5.2.3.1 Pre-muffled 25-mm, 0.45-micron Whatman glass fiber filters (GF/F), obtained from the laboratory.

5.2.3.2 Filter forceps—stainless steel, straight, flat, smooth tip, obtained from the laboratory.

5.2.3.3 25-mm polycarbonate filter holders for 0.20-micron GF/F filters, obtained from the laboratory.

5.2.3.4 Petri dish for particulate carbon samples, obtained from the laboratory.

5.2.3.5 40-mL glass vials for dissolved organic carbon samples, obtained from the laboratory.

6.0 Summary of Procedure

6.1 Pre-sampling Equipment Preparation

6.1.1 Prepare benthic flux chambers that may be used in either freshwater or marine waters (Figure 1). Sampling apparatus will include flexible tubing for each unit. Samplers will provide two peristaltic pumps and charged batteries at the onset of the field season, with flexible tubing and a connector with the sample tubing. Transport chambers to field staging area.

6.1.2 If benthic flux chambers, peristaltic pumps, batteries, or battery chargers are used by another organization without EAP staff, the organization must complete the EAP Equipment Loan form.

6.1.3 If using EAP Hydrolabs for continuous DO measurements within the chambers, complete pre-deployment maintenance and calibration according to SOP EAP033 (Swanson, 2010). If using other equipment, complete pre-deployment and maintenance and calibration in accordance with the QAPP. Oxygen measurements must be conducted in a continuously stirred vessel to avoid stratification within the chamber.

6.1.4 If Hydrolabs and stirrers are deployed by another organization without EAP staff, the organization must complete the EAP Equipment Loan form.

6.2 Benthic Flux Chamber Deployment

6.2.1 Refer to the QAPP for deployment locations, water depth, duration, and frequency of continuous and grab sample collection.

6.2.2 Transport chambers to each sampling location. Verify that Hydrolab or other DO sample recording equipment is firmly attached to benthic flux chambers and turn on the stirrer. Lower the chamber into the water column, initially with the open side up to remove all air bubbles. Invert the chamber near the bottom depth so

that the open side faces down. Be careful to fill the chamber with water from near the bottom of the water column and prevent the chamber from disturbing the sediment. Slowly lower the chamber into position using the chamber retrieval lines. Keep the sample collection tubing attached to the chamber with the open end out of the water. Avoid locations where the lower flanges are not in complete contact with the sediment, and avoid stirring up large volumes of sediment as the chamber is lowered into position. Collect the initial samples using methods described in Section 6.3. Following sample collection, disconnect the pump from the sample tubing, fold over the tubing and clamp to avoid contamination, and secure the end of the sample tubing to a float where it can be retrieved easily during the next sample round. Verify that all floats are functional before leaving the site.

6.3 Collecting Water Samples from Within Benthic Flux Chambers

6.3.1 Withdrawing Samples Using Peristaltic Pump

6.3.1.1 Refer to the QAPP for the sample collection schedule, including frequency and duration. The QAPP should specify the approximate length and inner diameter of tubing for each sample location, and the approximate volume necessary to clear the water from the tubing such that the tubing contains water from within the chamber for sample collection.

6.3.1.2 At each monitoring location, locate chamber tubing float. Carefully remove clamp from chamber tubing and attach to peristaltic pump or similar apparatus. Clear 1.5 times the inner volume of the tubing using a peristaltic pump or similar apparatus by pumping into a graduated container to verify volume pumped. Discard the cleared water.

6.3.1.3 If using an intermediate sampling container, rinse 1-L bottle three times with water cleared from the tubing, then continue to pump approximately 1 liter of sample water into the 1-L bottle for nutrient sampling.

6.3.1.4 Turn off the pump, disconnect the pump from the chamber tubing, fold the chamber tubing over and clamp to avoid contaminating water within tube, and reconnect chamber tubing to float to secure.

6.3.2 Dissolved Oxygen Samples

6.3.2.1 DO samples must be collected from the tubing directly as soon as possible and prior to processing nutrient samples to minimize the exchange of oxygen with the atmosphere. DO samples should be drawn into glass flasks without turbulence, in accordance with SOP EAP023 (Ward and Mathieu, 2013) for freshwater and SOP EAP027 (Bos et al., 2015) for marine waters. Bottles must be filled to the rim. Immediately after filling, add 1 mL of manganese chloride followed by 1 mL of sodium iodide-sodium hydroxide solution. Place the stopper on the rim and ensure that no bubbles are trapped inside. With a finger on the stopper, invert the

bottle to mix the reagents multiple times, and store in carrying case. Top each sample with deionized water.

6.3.3 Total Nutrient Samples

6.3.3.1 For total (unfiltered) nitrogen and phosphorus samples delivered to the laboratory, follow SOP EAP025 (Bos, 2013). In summary, rinse 20-mL graduated cylinders 3 times with sample water and discard. Using the 20-mL graduated cylinders, measure 20 mL of sample water and transfer to 60-mL total nutrients sample bottle. Graduated cylinders can be reused within a single field day. Alternatively, rinse syringe 3 times with sample water and discard. Use syringe to measure 20 mL of sample water into total nitrogen and phosphorus sample bottle.

6.3.3.2 For unfiltered nitrogen and phosphorus samples analyzed by other laboratories, consult with the laboratory for sample collection needs.

6.3.4 Dissolved and Particulate Nutrient Samples

6.3.4.1 For dissolved (filtered) nitrogen and phosphorus samples delivered to the laboratory, follow SOP EAP025 (Bos, 2013). In summary, rinse 60-mL syringe 3 times with sample water and discard. Attach 0.45-micron filter to syringe, filter 2 to 5 mL to waste, and then use filtered water to rinse the sample bottles 3 times and discard. Filter 100 mL of sample water directly into 175-mL dissolved nutrients sample bottle. Use one filter per sample, but syringes can be reused within a single field day.

6.3.4.2 For dissolved organic carbon samples delivered to the laboratory, follow SOP EAP025 (Bos, 2013). In summary, while the particulate carbon/nitrogen sample is being filtered using the 25-mm GF/F filters, collect 20-30 mL of the filtrate in a 40-mL glass vial.

6.3.4.3 For particulate carbon/nitrogen samples delivered to the laboratory, follow SOP EAP025 (Bos, 2013). In summary, filter sufficient sample through the 25-mm GF/F filters using a vacuum pressure of 5 to 7 psi to ensure a dark color on the filter pad; record the volume, up to 1 L. Place filter pad in labeled Petri dish and freeze. Use a single filter pad per sample, but syringes can be reused within a single field day.

6.3.4.4 For filtered nitrogen, filtered phosphorus, and particulate carbon/nitrogen samples analyzed by other laboratories, consult with the laboratory for sample collection needs.

6.3.5 Sample Labeling, Storage, and Delivery

6.3.5.1 Each sample must be labeled immediately following collection.

6.3.5.2 DO samples should be secured upright to avoid leakage, kept in the dark, and delivered to the EAP Operations Center (OC) within 24 hours of sample collection. EAP staff will analyze samples according to SOP EAP023 for freshwater and EAP027 for marine water.

6.3.5.3 Total and dissolved nutrient samples should be kept in coolers filled with ice and delivered to the EAP OC within 24 hours of sample collection. EAP staff will deliver samples to the appropriate laboratory in accordance with laboratory hold time requirements. Nutrient samples may be frozen for preservation in an upright position.

6.3.5.4 At the OC, EAP staff will transfer total nitrogen and phosphorus, dissolved nitrogen and phosphorus, particulate carbon/nitrogen, and DO samples to the freezer. EAP will deliver samples to the appropriate laboratory or transfer samples to the courier.

6.3.5.5 The QAPP will identify what laboratory will analyze the samples and what analytical methods are to be used. For nitrogen, phosphorus, and carbon samples submitted to the University of Washington's Marine Chemistry Laboratory, methods are described in SOP EAP025 (Bos, 2013).

6.4 Benthic Flux Chamber Removal

6.4.1 At the conclusion of the deployment period, collect final samples for dissolved oxygen and nutrient analyses using the procedures in Section 6.3. Using the retrieval lines with floats attached, gently pull the sampler from the sediment surface and lift through the water column. In deeper marine waters, this may require use of a winch or other hoist. Rinse the sampler free of sediment and pour out any remaining water from the chamber. Disconnect the Hydrolab or other DO monitoring device, turn off the stirrer, and return the chambers to EAP OC or other field staging area for cleaning, maintenance, or storage.

6.5 Benthic Flux Chamber Cleaning and Maintenance

6.5.1 Rinse and dry the benthic flux chambers and inspect for disconnected tubing or cracks. Inspect retrieval lines for abrasion. Replace any broken floats. Repair any leaks in the chambers. Any problems with the benthic flux chambers, peristaltic pumps, batteries, battery chargers, Hydrolabs, or stirrers must be reported to the EAP OC using the Equipment Problem Report Form.

6.6 Hydrolab Post-calibration, Downloading, Cleaning, and Maintenance

6.6.1 If using Hydrolabs owned by the Department of Ecology, deliver Hydrolabs to the EAP OC where EAP staff will perform post-deployment Hydrolab checks, download the data, and maintain the instruments using the SOP EAP033 Swanson, 2010).

6.6.2 If using other equipment, perform similar steps. Deliver pre- and post-deployment calibration information and data collected during the deployment to EAP staff in an appropriate electronic format, as described in the QAPP.

7.0 Records Management

7.1 Each sample collection event will be described in the field notebook with waterproof ink. At a minimum, field staff will record the date, time, location identification, sample laboratory identification number (if samples are submitted to Manchester Environmental Laboratory), analyses to be performed, and any ancillary data. Entries will be kept neat and concise.

7.2 Sample locations will be described in enough detail to locate on a United States Geological Survey 7.5-minute quad or an Environmental Information Management (EIM) System map. In marine areas or where other locators are not available, a global positioning system (GPS) unit will be used to record an accurate location using SOP EAP013. (Janisch, 2010). Coordinates will be recorded as per EIM requirements.

7.3 If samples are submitted to Manchester Environmental Laboratory, field staff will complete the Pre-sampling Notification form and Sample Container Request form prior to sampling. Field staff must submit the LAR form to accompany samples to the laboratory.

7.4 If samples are submitted to another laboratory, field staff will document appropriate sample information and follow sample handling requirements described in the QAPP.

8.0 Quality Control and Quality Assurance Section

8.1 Field staff will follow Quality Assurance/Quality Control procedures described in the QAPP. Field and laboratory replicate samples will be collected as directed in the QAPP.

9.0 Safety

9.1 All field staff must comply with the requirements of the EAP Safety Manual (EA Program, 2015). A full working knowledge of the procedures in Chapter 1 "General Field Work" is expected. If chambers are deployed in freshwater, field staff must comply with the section, "Working in Rivers and Streams." If chambers are deployed in marine waters, field staff must comply with the Chapter 2 section, "Operating Winches on Small Boats, Trailers, and Vehicles;" all onboard staff must be familiar with Chapter 3, "Boating."

9.2 For further field health and safety measures, refer to the EAP Safety Manual.

9.3 Canvas or leather gloves will protect hands from rope burns when lowering or raising sampling equipment from boats. Latex gloves should be worn to avoid bacterial or chemical exposure when extracting samples from chambers.

10.0 References

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